



Optimization of biodiesel yield using response surface methodology

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General Note

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ABSTRACT

Biodiesel produced from vegetable oils through transesterification is a common practice. The biodiesel yield through this process is affected by various process variables and contents of saturated and unsaturated fatty acids. In this study coconut and sunflower oils have been used and the yield has been optimized by using a Response Surface Methodology, considering molar ratio, catalyst concentration and reaction temperature as a process variable. The maximum yield with sunflower oil and coconut oil was obtained 95% and 77%, respectively with 6:1 molar ratio. Whereas the optimum value of catalyst concentration was found to be 1 wt% for sunflower oil and 1.25 wt% for coconut oil to get maximum yield. Also, the presence of the higher percentage of saturated fatty acids in coconut oil lead to low biodiesel yield.

Keywords: Catalyst concentration, Molar ratio, Temperature, Vegetable oil, saturated fatty acid and Unsaturated fatty acid.

1. INTRODUCTION

In the year 2008-09 India consumed about 51.7 million tonnes of petroleum diesel and this figure is estimated to exceed 153.4 million tonnes by 2030-31 (Reports, Govt. of India, 2010). Due to the increase of petroleum based fuel price in the past years and also increasing environmental concerns, Govt. of India has set the target on renewable energy utilization through a strategic national biofuels policy. In this policy, with the aim to achieve a target of 20% blending of petroleum diesel and bio-diesel by 2017 is proposed (N. P. B, Govt. of India, 2009). Consequently, India needs to produce about 30.68 million tonnes of biodiesel annually by the year 2030-31, to meet the target.

Biodiesel is a fuel produced from vegetable oil, animal fats and recycled oil from food industry (Yun-Hin et al., 2011) and chemically known as fatty acid methyl ester. Biodiesel is commonly produced by transesterification process. Transesterification is a reversible reaction, but in biodiesel production, the back reaction does not occur because glycerol formed is not miscible with the product. To produce 3 moles of biodiesel and 1 mole of glycerol, the stoichiometry of reaction is a 3:1 Mol ratio of alcohol to oil (Encinar et al., 2005; Gerpen, 2005). One of the most important physical properties of biodiesel fuel is viscosity. The acceptable kinetic viscosity range at 40 °C is 1.9-6.0 mm²/s as required by ASTM D6751 specification. The kinetic viscosity of fatty compounds (such as those found in biodiesel fuel) is influenced by the structure of the compound, chain length, the position, number and nature of double bonds and the nature of oxygenated moieties (Gryglewich, 2000; Kawashima et al., 2008; Rafaat, 2010). The high concentration of monoglycerides and diglycerides in biodiesel, not only affect the fuel quality, but also indicates an incomplete reaction during production. The presence of a catalyst accelerates the conversion of triglycerides to biodiesel (Meher, et al., 2004). Biodiesel is considered as renewable, biodegradable, environmentally benign, energy efficient and non detrimental to engine's operational performance (Van Gerpen, 2005). There is a number of factors or variables such as moisture content, reaction temperature, free fatty acid content, reaction time, oil to alcohol molar ratio, mixing intensity, type and amount of catalyst and co-solvent that can affect the production of biodiesel by the transesterification process in terms of yield and purity (Meher, et al., 2004; Demirbas, 2007; Jibrail, 2009; Sharma and Gupta, 2008). These variables usually have different effects on the transesterification process.

Biodiesel production is a costly and time consuming process and its yield depends on various parameters. It is, therefore, necessary to optimize the biodiesel production and for that the various modeling techniques are used by various investigators. Artificial Neural Network (ANN) tool was used by (Antonio et al., 2006) and Response Surface Methodology (RSM), has also been tested successfully by many researchers (Abdullah 2009; Jeong and Park, 2009; Ferella et al., 2010) to optimize the biodiesel yield considering various process variables such as the amount of catalyst, methanol, reaction time and temperature.

The main objective of this study is to optimize the biodiesel yield from the vegetable oils with dominating saturated fatty acids and with those where unsaturated fatty acids dominate. For this study sunflower oil and coconut oil were taken as samples because of the contents of variable amounts of unsaturated and saturated fatty acids present in them. This study reports the effect of fatty acid composition of oils and process variables on the biodiesel yield.

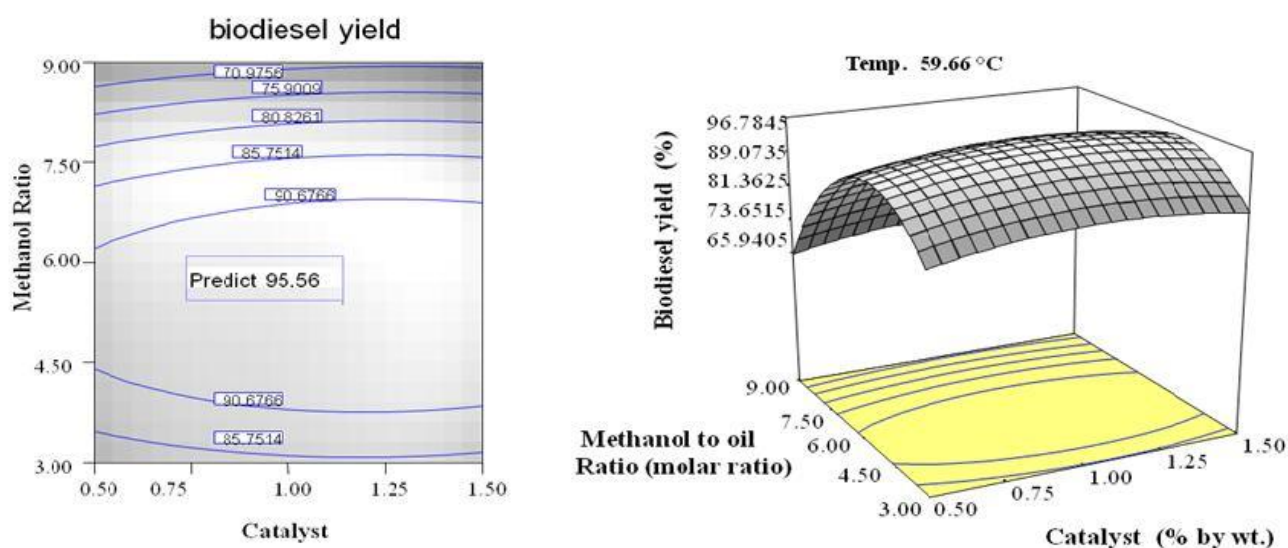


Figure 1

RSM plots of biodiesel yield vs Process parameter of sunflower oil

2. MATERIALS AND METHODS

2.1. Reagent and materials

The chemicals used in the experiments include analytical grade methanol (99%) and sodium hydroxide pellets. The crude oils of sunflower and coconut used in the study were purchased from Jatropha Vikas Sansthan, Idgah Circle, Delhi. The key factors affecting the production of biodiesel in terms of production yield and purity of biodiesel include reactant purity, reaction time, reaction temperature, catalyst type and their concentration and molar ratio of alcohol to oil. Also, the process variables and operating conditions affect the biodiesel yield and Physio chemical properties depends upon the type of feedstock. For this study oils were selected on the basis of the amount of saturated and unsaturated fatty acid contents. Among the selected oils, the coconut oil has a higher concentration of saturated fatty acids, whereas the sunflower contains large amount of unsaturated fatty acids.

2.2. Trans-esterification Procedure

The batch reaction kinetics experiment was employed and experiments were performed on 1000 ml batch reactor with hot plate having a thermostat to maintain the desirable temperature and magnetic stirrer to stir the mixture. One liter of sample vegetable oil was mixed with required amount of methanol and sodium hydroxide (methoxide). The mixture was poured into a three necked glass flask mounted with condenser and thermometer. The condenser was used to recover the escaping methanol from the flask during the reaction. A magnetic stirrer was used to heat the solution at required temperature. After the end of the reaction, the mixture was cooled down to room temperature and transferred to a separating funnel. The reaction mixture was then allowed to settle for a minimum of 8 hours. Now a clear separation of two layers with the top layer as ester and glycerin settling at the bottom was observed due to its higher specific gravity. The two layers were then separated and ester was washed three times with lukewarm water to remove any traces of methanol and sodium hydroxide etc. Ester was heated again to remove the moisture, if any.

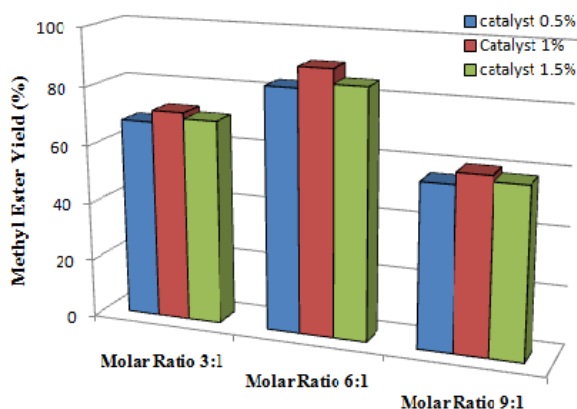


Fig. 2 Effect of molar ratio of methanol to oil and catalyst on methyl ester yield at reaction temperature 30 °C

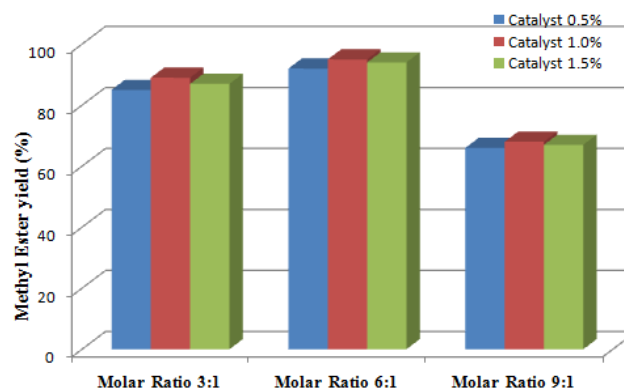


Fig 3 Effect of molar ratio of methanol to oil and catalyst on methyl ester yield at reaction temperature 60 °C

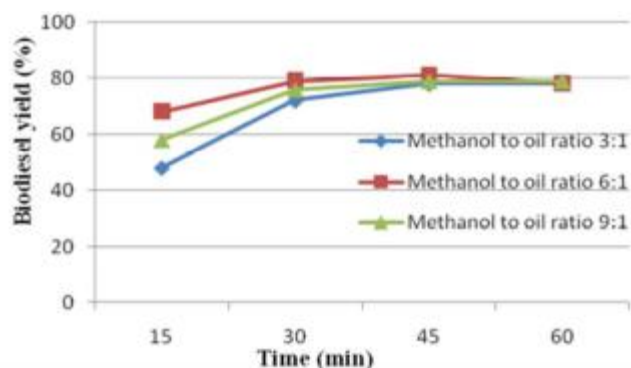


Figure 4

Effects of Time and molar ratio of methanol to oil on biodiesel yield

2.3. Experimental Design

For sunflower oil a three level, three factors and for coconut oil three levels, two factor central composite designs were employed to determine the optimum conditions. The reaction temperature, methanol to oil ratio and the amount of catalyst were independent variables selected and the levels of the variable were coded as -1, 0 and +1. The range of the operating conditions and the experiments planned are clearly demonstrated in table 1 and 3. The dependent variable (response) was taken as percentage biodiesel yield (Response) which was determined depending on the result of GC analysis as below:

$$\text{Yield} = 100 - \sum (\text{triglycerides} + \text{diglyceride} + \text{monoglyceride} + \text{glycerin})$$

2.4. Experimental Analysis

The experimental result obtained were analyzed by the response surface regression methodology, using the following second order polynomial equation

$$Y = \beta_0 + \sum_{k=1}^n \beta_k X_k + \sum_{k=1}^n \beta_{kk} X_k^2 + \sum_{k=1}^n \sum_{l=1}^n \beta_{kl} X_k X_l$$

Where Y represents the dependent variable i.e. percentage biodiesel yield; X_k and $X_k X_l$ are the terms of the main effects and the interaction effects, respectively. A description of the experimental design and the relationship between the coded and the real values of the variables given below:

= Value of variable – midpoint value of variable k / Range of Values of variable k

X_k

Table 1

Experimental Results of Sunflower oil

Batch	Catalyst Concentration (Wt. %)	Methanol: Oil Ratio (Molar Ratio)	Temp. (DegC)	Biodiesel Yield (%)
1	0.5	3:1	30	68
2	1	3:1	30	72
3	1.5	3:1	30	70
4	0.5	3:1	60	85
5	1	3:1	60	89
6	1.5	3:1	60	87
7	0.5	6:1	30	83
8	1	6:1	30	90
9	1.5	6:1	30	85
10	0.5	6:1	60	92
11	1	6:1	60	95
12	1.5	6:1	60	94
13	0.5	9:1	30	56
14	1	9:1	30	60
15	1.5	9:1	30	58
16	0.5	9:1	60	66
17	1	9:1	60	68
18	1.5	9:1	60	67
19	0.5	6:1	50	91
20	1	6:1	60	92

Table 2

Analysis of Variance table and factor effects of Sunflower oil

Factor	Factor Effect	Sum of Square	Deg. of Freedom	Mean Square	F Value
A	0.92	10.08	1	10.08	2.47
B	-6.37	92.89	1	92.89	22.72
C	5.61	566.72	1	566.72	138.63
A ²	-3.08	38.03	1	38.03	9.30
B ²	-17.71	716.77	1	716.77	175.34
C ²	-0.55	0.099	1	0.099	0.024
AB	-0.13	0.13	1	0.13	0.031
AC	-0.083	0.083	1	0.083	0.020

BC	-2.00	48.00	1	48.00	11.74
Residual		36.79	9	4.09	
Total		3344.80	19		

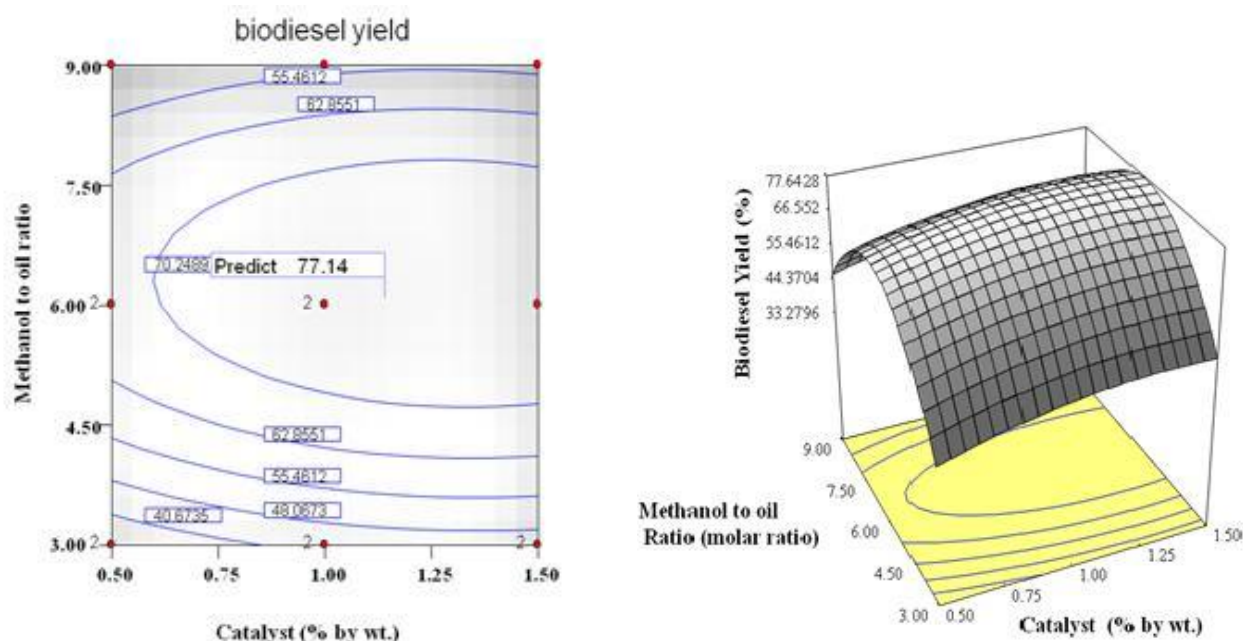


Figure 5

RSM plots of biodiesel yield vs Process parameter of coconut oil

The design expert version 6.0 was used for regression analysis of variance ANOVA. Response surfaces were developed using the fitted polynomial equation obtained from the regression analysis.

3. RESULT AND DISCUSSION

3.1. Sunflower Oil

RSM has been used successfully for optimization of biodiesel production from sunflower and coconut oil. The final response model equation for sunflower oil by which the synthesis of biodiesel was estimated is as follows.

$$Y = 90.82 + 0.92*A - 6.37*B + 5.61*C - 3.08*A^2 - 17.71*B^2 - 0.55*C^2 - 0.13*A*B - 0.083*A*C - 2.00*B*C$$

Whereas:

A = catalyst concentration

B = molar ratio

C = reaction temperature

The model co-efficient and probability values are shown in table 2. The optimum condition for the transesterification of sunflower oil is estimated as catalyst 1.06 wt%, methanol to oil ratio 5.82:1, temperature 59.10 °C and the predicted fatty acid methyl ester content was 96.08%.

RSM is illustrated with 2 and 3- Dimensional plots by presenting the response in function of three factors keeping the other constant. Figure 1 shows the yield of biodiesel in relation to the methanol to oil ratio, catalyst concentration and reaction temperature. Plots (Figure 1) show that methanol to oil ratio has a direct effect on the yield of methyl ester and maximum yield was obtained at 1% catalyst concentration, 5.82:1 molar ratio and reaction temperature 60 C. Any further increase or decrease in the molar ratio affects the biodiesel yield adversely.

The result obtained from experimental values for biodiesel yield at different molar ratio and catalyst concentration are represented in the form of a bar graph. Figure 2 shows the methyl ester yield for given molar ratio and catalyst concentration at 30 °C reaction temperature. It is referred from the figure that maximum yield was obtained at 6:1 methanol to oil ratio and 1% catalyst concentration. The similar trends are seen in figure 3, for given molar ratio, catalyst concentration and reaction temperature. It is also inferred from the figure 3 that at 60°C reaction temperature maximum yield can be achieved at 6:1 molar ratio and 1% catalyst concentration.

Table 3

Experimental Results of Coconut oil

Batch	Catalyst Concentration (Wt. %)	Methanol: Oil Ratio (Molar Ratio)	Biodiesel Yield (%)
1	0.5	3:1	32
2	1	3:1	40
3	1.5	3:1	42
4	0.5	6:1	60
5	1	6:1	77
6	1.5	6:1	75
7	0.5	9:1	45
8	1	9:1	55
9	1.5	9:1	56
10	0.5	3:1	35
11	1	3:1	45
12	1.5	3:1	46
13	0.5	6:1	74
14	1	6:1	76

Table 4

Analysis of Variance table and factor effect of Coconut oil

Factor	Factor Effect	Sum of Square	Deg. of Freedom	Mean Square	F Value
A	4.58	166.52	1	166.52	17.86
B	5.62	248.34	1	248.34	26.63
A ²	-3.73	43.24	1	43.24	4.64
B ²	-27.90	2275.57	1	2275.57	244.03
AB	-0.90	4.17	1	4.17	0.45
Residual		65.27	7	9.32	
Total		3305.71	13		

3.2. Coconut Oil

It is reported in the literature that during the transesterification the reaction time between 10-25 minutes is crucial after that its impact reduces considerably. The facts were established during the transesterification of sunflower oil. The experimental results obtained with different methanol to oil ratios, reaction temperature and catalyst concentration with sunflower oil concluded that after 30-35 minutes the methyl ester yield (%) curve level-off, as shown in Figure 4. Therefore, for the esterification of coconut oil the reaction time was kept constant and assigned the value of 60 minutes. The final response model equation for coconut oil by which the synthesis of biodiesel was estimated is as follows. $Y = 76.01 + 4.58A + 5.62B - 3.73A^2 - 27.90B^2 - 0.90AB$

The model co-efficient and probability values are shown in table 4. The optimum condition for the transesterification of coconut oil at reaction temperature 60°C is estimated as catalyst 1.26 wt%, methanol to oil ratio 6.12:1. The predicted value of fatty acid methyl ester was obtained 77.50% for given values of process variables.

RSM is illustrated with 2 and 3- Dimensional plots by presenting the response in function of two factors keeping the other constant. It is visualized by the yield of biodiesel in relation to the methanol to oil ratio and catalyst in Figure 5. The figure shows that methanol to oil ratio has a direct effect on the yield of methyl ester and optimum yield was obtained at 1.26% catalyst

concentration and 6.12:1 molar ratio. Further increase or decrease of the values of catalyst concentration and molar ratio affects the biodiesel yield adversely.

In order to authenticate the results of RSM technique the experiments were carried out at reaction temperature 60 °C and taking molar ratio 6:1 for both the oils, whereas, catalyst concentration was taken as 1 wt% and 1.25% for sunflower oil and coconut oil, respectively. The yield of biodiesel obtained with the sunflower oil and coconut oil was 95% and 77%, respectively, which is almost in the range of theoretical values and hence authenticates the sanctity of the technique.

4. CONCLUSION

RSM assisted technique for optimization of process parameter to get the maximum biodiesel yield from sunflower and coconut oil found very promising. For both the oils, the maximum yield was obtained at a molar ratio for oil to methanol of 6:1 and reaction temperature 60 °C. However, the optimum value for catalyst concentration was found to be 1 wt % for sunflower oil and 1.25 wt% for coconut oil. Further increase in molar ratio and catalyst concentration causes adverse effect on the yield of biodiesel. The yield of methyl esters of sunflower and coconut were obtained of the order of 95% and 77%, respectively at optimum values of process parameter. However, the low yield of ester with coconut oil is attributed to the large percentage of saturated fatty acids present in the oil.

DISCLOSURE STATEMENT

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